

## PATENT SPECIFICATION

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## COMPLETE SPECIFICATION

## Improvements relating to Processes for the Production of Cellulose Pulp

We, ENGLISH CELLULOSE DERIVATIVES LIMITED, a British Company, of Church Street East, Radcliffe, in the county of Lancaster, and GUSTAV ULLMANN, of

5 Austrian nationality, of 81, Singleton Road, Kersal, Salford, in the county of Lancaster, England, do hereby declare the nature of this invention and in what manner the same is to be performed, to

10 be particularly described and ascertained in and by the following statement:—

This invention relates to the production of cellulose pulp from fibre-bearing vegetable materials.

15 An object of the invention is to provide a process which makes use of the effluents from the pulping of cellulosic materials and thereby contributes towards the solution of the problem of their disposal.

20 The process of the present invention makes use of various kinds of effluents produced in the pulping of cellulosic material to pulp an entirely fresh batch of cellulosic material.

25 The effluents obtained from the pulping of cellulosic materials are generally speaking of three different kinds namely:—

(1) The effluents from the soda process, 30 which is also known as the "caustic process". This process is mainly used for pulping plants such as cereal straw or the like, and more rarely for pulping wood to produce a pulp having special

35 properties.

(2) The effluents from the sulphate process, which is mainly used for the pulping of wood by boiling with a solution containing principally caustic

40 soda and sodium sulphide.

(3) The effluent from the sulphite process, which effluent contains calcium bisulphite, magnesium bisulphite, sodium bisulphite, etc.

45 In all these processes, after finishing the boil, the liquor now called "black liquor" is drained away from the pulp. The pulp is washed and the wash water is added to the black liquor. These effluents 50 contain on the average about 50% or more of the organic matters in the original plant.

The very great difficulties due to the content of these organic matters in the effluents are well known and are a worldwide problem. 65

Usually these effluents, especially those from the process (1) and (2) above, are concentrated in an evaporator plant to a liquor of high viscosity, which is finally

60 burnt in a furnace, whereby the alkali contained in it is transformed into soda ash, which later on is regenerated to caustic soda by well known caustification methods. In this case the organic

65 matters contained in the concentrated effluents supply, when burning almost all the heat which is necessary in the furnace, so that this plant is to a large extent self-sufficient as regards heat. But the fuel

70 value of the organic matters in these effluents is not sufficiently large to deal in the recovery plant with the more dilute effluents without an additional supply of coal or other fuel; this, however, makes

75 the cost prohibitive and, therefore, very large quantities of wash waters in which the total content of organic matters is still

relatively high have to be released into the water courses and are responsible for

80 great difficulties owing to the pollution caused thereby, and inorganic materials are also lost, which otherwise would increase the efficiency of the recovery plant. 85

The position is also unsatisfactory as far as sulphite effluents are concerned, which have an acid reaction.

According now to the present invention, raw cellulosic fibre-bearing vegetable

90 material is first subjected to a pre-treatment with a dilute solution of an acid or an acid salt and the pre-treated material is digested with the alkaline effluent from an alkaline pulping process

95 which may be either the caustic soda or the sulphate process. The dilute acid solution may consist either of a solution of fresh acid or of an acid salt or a mixture thereof or of waste sulphite

100 effluent. In the latter case the pH of the sulphite black liquor, which is usually in the region of 3—4, is reduced to about 1.0—1.5 by the addition of an acid or acid

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salt. The alkaline solution used in the second stage of the process advantageously consists of black liquor from a boil with caustic alkali, which liquor may be 5 undiluted or slightly diluted.

Owing to the pre-treatment with the acid solution or waste sulphite liquor, it is found that it is much easier to obtain a satisfactory pulp by boiling the pre-treated material with the waste alkaline effluent liquor, which although the concentration of active alkali is much reduced as compared with that of the initial boiling liquor, still contains some 10 unconsumed alkali. The effluent, however, being costless can be used in a larger ratio than usual with all the well-known advantages attendant thereon.

Normally the boil of cellulosic material 20 is made in a ratio of 1 of cellulosic material to about 4-5 of liquor. The first liquor drained away after the boil is very strong, but if mixed with wash waters of different kinds, it becomes 25 dilute and much more of this liquor is then available, allowing a very high ratio of liquor to cellulosic material to be used. If this is done the alkali then present is sufficient to effect successful pulping.

It should be mentioned that straw and the like on the one hand and wood and the like on the other hand need to be boiled in liquors of very different concentrations, that for wood being the stronger. Therefore, the alkaline effluents from the treatment of straw are less concentrated than those from pulping of wood. Consequently it is less economical to use effluents from straw even at a higher pressure for dealing with wood-like materials to produce 40 a fresh lot of pulp therefrom. But as a consequence of the acid pre-treatment, it is made easier and more economical to use such effluents from a straw boil for pulping a fresh lot of straw or similar cellulosic materials. Similarly, effluents from a wood boil can be very advantageously used for pulping straw and similar materials, and they are very suitable also 45 for dealing with wood owing to their higher concentration, particularly if the fresh material is boiled with them at a higher pressure, and, if necessary, for a longer period.

55 The process of the invention may be carried out in various ways.

One method consists in pre-treating a fresh batch of straw with a dilute acid solution or a waste sulphite effluent and 60 boiling the pre-treated material with or without pressure with undiluted or slightly diluted black liquor from a straw pressure boil. Straw is normally boiled with an alkaline liquor of a strength such 65 that the effluent therefrom, i.e., the black

liquor, has a density of say 14° Tw. This black liquor is strong and active and if used even without pressure for pulping a fresh batch of straw which has been pre-treated as described, a pulp of high 70 quality as regards cellulose content and cleanliness may be obtained.

When a higher class pulp is required, the same liquor of 14° Tw. may be used for boiling a fresh lot of pre-treated straw 75 under about 50-60 lbs./sq. in. pressure for 3-4 hours, in a ratio of for instance, 1:8 or 1:10; this relatively mild treatment results in a pulp of very good qualities for many purposes, such as for fine 80 papers, the pulp having a very good shade after bleaching and the yield being remarkably high.

Alternatively the black liquor after being diluted with wash water, when it 85 has a density of say 4-5° Tw. is used for pulping straw which has been pre-treated with a fresh acid solution or sulphite effluent when a useful pulp is produced. The quality of the pulp obtained can be 90 improved if the boil is made in two stages instead of one stage and preferably in a continuously working apparatus, even without pressure and with a short boiling time of say 2-3 hours for each stage. 95

It is possible to manufacture much larger quantities of pulp using weaker solutions if the pulp is to be used for special purposes, for which the highest cleanliness, a very bright shade after 100 bleaching, and the largest possible cellulose content are not necessary for instance for boards and newsprint.

By varying the concentration of the liquor, boiling times, ratio and other conditions very great variations in the pulp 105 produced can be made.

In the processes described no fresh alkali is added to the alkaline effluent used and the effluent from the second boil 110 becomes very economical in organic matters which provide a large excess of heat in the furnace of the recovery plant, which excess of heat can be usefully employed for various purposes. 115

In order that the invention may be easily understood and readily carried into effect, the following examples are given.

#### EXAMPLE 1.

Straw is pre-treated for two hours in a 120 continuous way with a boiling solution of sodium bisulphite of 1½% concentration and in a ratio of 1:8. The pre-treated material is washed and boiled; the ratio being 1:10, for three hours in a continuously working apparatus with wash water of 8° Tw. density from a straw boil in which the straw was boiled for five hours at a pressure of 70 lbs. per square 125

inch with 20% caustic soda based on the raw straw weight, the ratio being 1:4½.

**EXAMPLE 2.**

Straw is pre-treated by boiling for two 5 hours in a continuous apparatus with sulphite effluent having a density of 12° Tw. the pH of which has been reduced to 1—1.5 by the addition of acid, the ratio being 1:8.

10 The pre-treated material is washed and boiled for three hours, again in a continuous apparatus, with black liquor (ratio 1:10) diluted to a density of 9° Tw. from a straw pressure boil which was 15 made under the same conditions as the straw pressure boil of Example 1. Black liquors and wash waters from this boil are sent to the recovery plant. The pulp is finally 20 bleached after washing in the usual way.

We are aware that it has previously been proposed to treat wood pulps, for example, sulphite pulps and soda pulps with an alkaline solution which already 25 contains substances resulting from the treatment of ligno cellulosic material with an alkali or an alkali reagent and we make no claim thereto. The present invention is distinguished therefrom in that 30 the strength of the acid or acid salt solution used for the pre-treatment is such that only relatively small amounts of impurities are chemically changed or removed from the raw cellulosic material 35 as compared with the amounts removed in producing pulp, for the purpose of enabling a subsequent normal digestion process to be carried out more easily and effectively.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. A process for the production of pulp 45 from raw cellulosic vegetable materials according to which the raw cellulosic material is pre-treated with a dilute solution of an acid or an acid salt and the pre-treated material is digested with 50 the alkaline effluent from an alkaline pulping process.

2. A process according to claim 1, in which the acid solution used for the pre-treatment is waste sulphite effluent. 55

3. A process according to claim 2, in which the pH of the waste sulphite effluent is reduced to about 1—1.5 before being used by adding an acid or an acid salt. 60

4. A process according to any preceding claim, wherein the alkaline effluent from a boil of wood or straw or the like is used for the digestion of wood or straw or the like.

5. A process according to any preceding claim wherein the alkaline effluent from a boil of wood or straw or the like is used for the digestion of straw or the like. 65

6. A process for the production of pulp 70 from fibre-bearing vegetable materials substantially as described and in accordance with any of the preceding examples.

Dated this 7th day of March, 1945.

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